726165

NAVAL AIR DEVELOPMENT CENTER

Warminster, Pennsylvania

REPORT NO. NADC-MA-7066   8 June 1971

A COMPARISON OF VARIOUS TEST METHODS FOR DETECTING HYDROGEN EMBRITTLEMENT

AIRTASK A3205203/202B/1F31541201
WORK UNIT-OD

Approved for public release; distribution unlimited.

UNCLASSIFIED
Security Classification

DOCUMENT CONTROL DATA - R & D

(Security classification of title, body of abstract and indexing annotation must be entered when the overall report is classified)

1. ORIGINATING ACTIVITY (Corporate author) AERO MATERIALS DEPARTMENT NAVAL AIR DEVELOPMENT CENTER WARMINSTER, PA. 18974	2a. REPORT SECURITY CLASSIFICATION UNCLASSIFIED 2b. GROUP NONE

3. REPORT TITLE
A COMPARISON OF VARIOUS TEST METHODS FOR DETECTING HYDROGEN EMBRITTLEMENT

4. DESCRIPTIVE NOTES (Type of report and inclusive dates)
PROGRESS REPORT

5. AUTHOR(S) (First name, middle initial, last name)
E. J. JANKOWSKY

6. REPORT DATE	7a. TOTAL NO. OF PAGES	7b. NO. OF REFS
8 June 1971	20	2

8a. CONTRACT OR GRANT NO. b. PROJECT NO. AIRTASK A3205203/202B/1F51541201 c. Work Unit OD d.	9a. ORIGINATOR'S REPORT NUMBER(S) NADC-MA-7066 9b. OTHER REPORT NO(S) (Any other numbers that may be assigned this report) NONE

10. DISTRIBUTION STATEMENT
APPROVED FOR PUBLIC RELEASE; DISTRIBUTION UNLIMITED.

11. SUPPLEMENTARY NOTES	12. SPONSORING MILITARY ACTIVITY
NONE	NAVAL AIR SYSTEMS COMMAND DEPARTMENT OF THE NAVY WASHINGTON, D. C. 20360

13. ABSTRACT

Four hydrogen embrittlement test methods were evaluated using three paint strippers as the embrittling media. Results were compared with those obtained with notched C-rings, the method now prescribed in paint stripper specifications. In general, all the methods give good results and good correlation. The main differences were in ease of use.

14 KEY WORDS	LINK A		LINK B		LINK C	
	ROLE	WT	ROLE	WT	ROLE	WT
Hydrogen Embrittlement Testing						
High Strength Steels						
Paint Removers						

DEPARTMENT OF THE NAVY
NAVAL AIR DEVELOPMENT CENTER
WARMINSTER, PA. 18974

Aero Materials Department

REPORT NO. NADC-MA-7066 8 June 1971

A COMPARISON OF VARIOUS TEST METHODS FOR DETECTING HYDROGEN EMBRITTLEMENT

AIRTASK A3205203/202B/1F51541201
WORK UNIT-OD

Four hydrogen embrittlement test methods were evaluated using three paint strippers as the embrittling media. Results were compared with those obtained with notched C-rings, the method now prescribed in paint stripper specifications. In general, all the methods give good results and good correlation. The main differences were in ease of use.

REPORTED BY: E. J. Jankowsky
E. J. JANKOWSKY
Chemical Metallurgy Branch

APPROVED BY: S. J. Ketcham
S. J. KETCHAM, Head
Chemical Metallurgy Branch

F. S. Williams
F. S. WILLIAMS, Superintendent
Metallurgical Division

SUMMARY

INTRODUCTION

For some years the Naval Air Development Center has used sustained load tests with notched C-rings or notched tensile bars to evaluate hydrogen embrittlement damage to high strength steels. These specimens are sensitive and give reproducible results but are expensive to machine and require long testing times.

Several instrumental methods have been developed in the past several years. Two involve the use of probes, the third utilizes metal strips. A comparative study of three of these has been completed and results correlated with those obtained from notched C-rings. A notched bolt with a miniature calibrated proving ring was also included in the investigation. Paint strippers were used as the embrittling media largely because MIL-R-81294A (reference (a)) now contains an embrittlement test using notched C-rings.

This work was performed under Work Unit F51 541 201 OD as part of the continuing effort to find better means of measuring hydrogen embrittlement resulting from pickling, electroplating and related processing.

SUMMARY OF RESULTS

Results of the three instrumental methods and the two notched specimens showed excellent correlation. One of the paint strippers used is very embrittling, the second somewhat less so and the third is not embrittling. All the methods were as effective in assessing the degree of embrittlement of each paint stripper as was the notched C-ring.

CONCLUSIONS

The three instrumental methods varied in complexity of use. The method utilizing strips was the simplest to use. The notched bolt with the calibrated proving ring, however, is the simplest test to conduct and the bolts and rings are readily available, but at the present time there is only one source of supply.

RECOMMENDATIONS

It is recommended that consideration be given to preparation of a Federal Test Method Standard for Hydrogen Embrittlement Testing which would include the notched C-ring and the other methods covered in this report.

TABLE OF CONTENTS

EXPERIMENTAL PROCEDURE

Three paint stripping solutions were used for this evaluation. The strippers were classified very embrittling, moderately embrittling, and non-embrittling on the basis of results obtained with notched C-rings.

The test method used for notched C-rings consisted of cadmium plating the outside surface of the rings in a cyanide bath without brighteners (50 asf, 2 min), baking for 24 hours at 375°F, stressing to 75% of the breaking strength, and then dipping the closed half of the ring into the paint stripper. Immersion time was approximately 1 minute. Stripper was allowed to remain on the ring surface with no rinsing after dipping. Time to failure was measured with electric timers attached to microswitches as shown in Figure 1.

Drilube Notch Bolts were prepared and treated in the same manner as the C-rings, except that pre-plate cleaning included grit blasting with 120 grit aluminum oxide and baking time after plating was 1 hour in the oven furnished with the equipment. Time to failure was determined by visual examination at intervals, or in some cases, by an audible "snap" when the bolts broke.

Drilube bolts are supplied with a miniature proving ring calibrated to apply a 75% NTS stress to a bolt passing through its diameter. The ring is compressed in a hydraulic loading device with the bolt in place. When the ring has been compressed a predetermined distance (furnished by the manufacturer), a small collar is swaged onto the bolt and the load on the hydraulic jack is released, leaving the bolt in tension. Each bolt comes with its own calibrated ring. Bolts are made of 4340 steel, heat treated to the 260 - 280 KSI strength level. The notch is 60° with a 0.005" radius.

The HEP Tester consists of timing devices attached to banks of bent strip holders. Five steel strips are used for each test, and the time for three strips to break is recorded as the "median" failure time. Strips are ½" X 7½" X .022" thick, with double tabs stamped through the center to act as notches. The stress applied is predetermined by the distance between the restraining ends on the tester. The ends of the strips are simply squeezed by hand until they can be popped into the tester. The design of the HEP Tester does not permit the testing of liquids or slurries with the specimens under stress. It was therefore necessary to alter the procedure used for testing C-rings.

HEP strips were plated with unbrightened cyanide cadmium for 2 minutes at 50 asf, baked 24 hours at 375°F, immersed in stripper for 6 hours, washed in running water followed by acetone, and immediately placed under stress in the timing device.

The Lawrence Hydrogen Detection Gauge makes use of a steel walled tube, similar to a 6V6 tube, as a hydrogen probe. The quantity of hydrogen passing through the wall of the tube during plating or processing is measured electronically and displayed on a hydrogen index scale. The maximum value measured after the tube is placed in the built-in oven is called the heat peak, H_p, and the time for the hydrogen index to decrease to one half the heat peak value is called lambda, λ. The heat peak is related to the amount of hydrogen introduced into the tube and lambda is a measure of the internal clean-up and outward diffusion of hydrogen.

Tubes with the entire end section grit blasted immediately prior to use were used for the measurement of embrittlement by paint strippers. A blast cleaning unit especially designed for grit blasting Lawrence tubes, 120 grit dry aluminum oxide, and a total blasting time of 30 ± 2 sec. were used for the tube cleaning. Prior to blasting, the tubes had been baked out on a special bake-out rack to remove residual hydrogen. The cleaned tubes were immediately plated with unbrightened cyanide cadmium at 50 amps/sq. ft. for 2 minutes and again baked to remove hydrogen. The plated section of the tube was immersed in stripper for 6 hours, taken out, washed in water and acetone, connected to the probe socket of the Gauge, and the heat peak and lambda value measured.

A much more detailed description of the procedure is given in reference (b).

The Drilube Hydrogen Gauge employs standard 6V6 tubes with steel walls. Blast cleaning is similar to that used for the Lawrence Gauge, but is considered less critical and is not timed. Following grit blasting, each tube is calibrated in a solution containing 50 gms/liter NaCN and 50 gms/liter NaOH. Tubes were reblasted following calibration, plated in an unbrightened cyanide cadmium bath, baked to remove hydrogen, and immersed in stripper for 6 hours. The stripper was cleaned off with water and acetone, and the heat peak and lambda values measured from the recorded curve of hydrogen index versus time.

The embrittling characteristics of paint strippers often change with age as demonstrated in reference (b). Care was taken to run the various tests simultaneously to avoid differences caused by chemical changes in the stripper.

RESULTS

Results of the various tests are given in Table I.

Cadmium plated and baked, notched C-rings are required to sustain a load equal to 75% of their breaking strength for 200 hours after immersion in paint stripper, according to reference (a). Stripper A caused failure of four specimens in times ranging from 1.3 to 1.9 hours. A fifth specimen broke in 34.4 hours. Eight specimens were tested in Stripper B with failure times ranging from 3.0 to 34.9 hours for seven specimens and one specimen not failing in 200 hours. Four notched C-rings tested in Stripper C, a non-embrittling stripper, did not fail in 300 hours.

All other types of specimens were cadmium plated and baked prior to immersion in paint stripper for comparison with C-rings.

HEP Strips, heat treated to a Rockwell C hardness of 51, broke in a median time of 13.1 hours after immersion in Stripper A, 35 hours after immersion in Stripper B, and did not fail after 500 hours in Stripper C. (Median time is taken as the time the third strip in a series of five breaks).

Standard HEP strips, which are heat treated to a lower strength level than those described above, were not sensitive enough to show embrittlement in the paint strippers tested. Two specimens broke within the 200 hour testing time, but the third "median" strip, on which failure time is based, did not fail.

Four Drilube Notch Bolts broke in times ranging from 1.3 to 2.4 hours in Stripper A. Six bolts, tested in Stripper B, broke in times between 7 and 100 hours. Exact times were not obtained for this group because they broke during a long weekend shutdown and no timing device is used with the bolts. The four bolts tested in Stripper C did not fail in 500 hours.

Heat peak (HP) values above 1 are generally considered beyond the "safe" hydrogen level for high strength steels. Lambda (λ) values are less important for paint strippers because they are an indication of the permeability of the plated coating rather than a measure of the amount of hydrogen introduced. Stripper A gave HP values of 212 and 207, Stripper B, 4.2 and 6.8, and Stripper C, 0.23 and 0.23, in good agreement with notched C-ring results.

The Drilube HP values should be lower than 1 on the 7 scale in order to be considered safe. Much higher values were obtained for Stripper A, slightly higher for Stripper B. Stripper C was not reported because the masking undercut badly during the tests. However, the values obtained, even with the oversized area exposed, were considerably below the hydrogen limit for safety.

D I S C U S S I O N

Any of the methods tried appears to be satisfactory for the testing of paint strippers, although each one has some undesirable features.

The Lawrence Hydrogen Detection Gauge shown in Figure 2 gives reproducible results that are very useful for determining the extent of embrittlement induced by paint strippers or electroplating processes. Its principal disadvantage appears to be the extraordinary care that must be taken in each step of probe preparation and measurement in order to obtain reproducible results. This disadvantage still allows the Gauge to be a useful laboratory tool, but limits its usefulness as a production or shop type measuring device. A more comprehensive investigation of this instrument was reported in reference (b).

The Drilube Hydrogen Evaluation Gauge, Figure 3 , a device similar to the Lawrence Gauge, appears to be somewhat simpler in probe preparation and measurement than the Lawrence Gauge. However, at this time, the measurement of embrittlement by paint strippers is still in the experimental stage and definite procedures have not been worked out. Its most useful function appears to be the determination of embrittlement from plating processes, and especially, the prediction of the efficacy of baking for relieving embrittlement.

The HEF Tester shown in Figure 4, is a potentially excellent method for determining embrittlement. Its chief fault, at present, is that the standard strips are not sensitive enough to fail under slightly embrittling conditions. This fault is being corrected by the manufacturer and the new, R_C 51 hardness strips appear to be much more sensitive. The principle advantages to this method are that it is extremely simple, very low in cost, and adaptable to shop use.

The Drilube Notched Bolt shown in Figure 5 is considered to be ready for use as it stands. Bolts may be obtained from the manufacturer in any desired quantity at a relatively low cost, and the loading equipment is simple and inexpensive. The only fault this method has is that at present there is no timing device to measure failure times,and failures must be determined by visual examination at convenient intervals. It should not be difficult, however, to modify the equipment to include timers.

REFERENCES

(a) Specification MIL-R-81294A, Remover, Paint, Epoxy System

(b) Naval Air Development Center Report No. NADC-MA-7052, Evaluations of the Lawrence Hydrogen Detection Gauge for the Prediction of Hydrogen Embrittlement, Progress Report, 9 October 1970

TABLE I

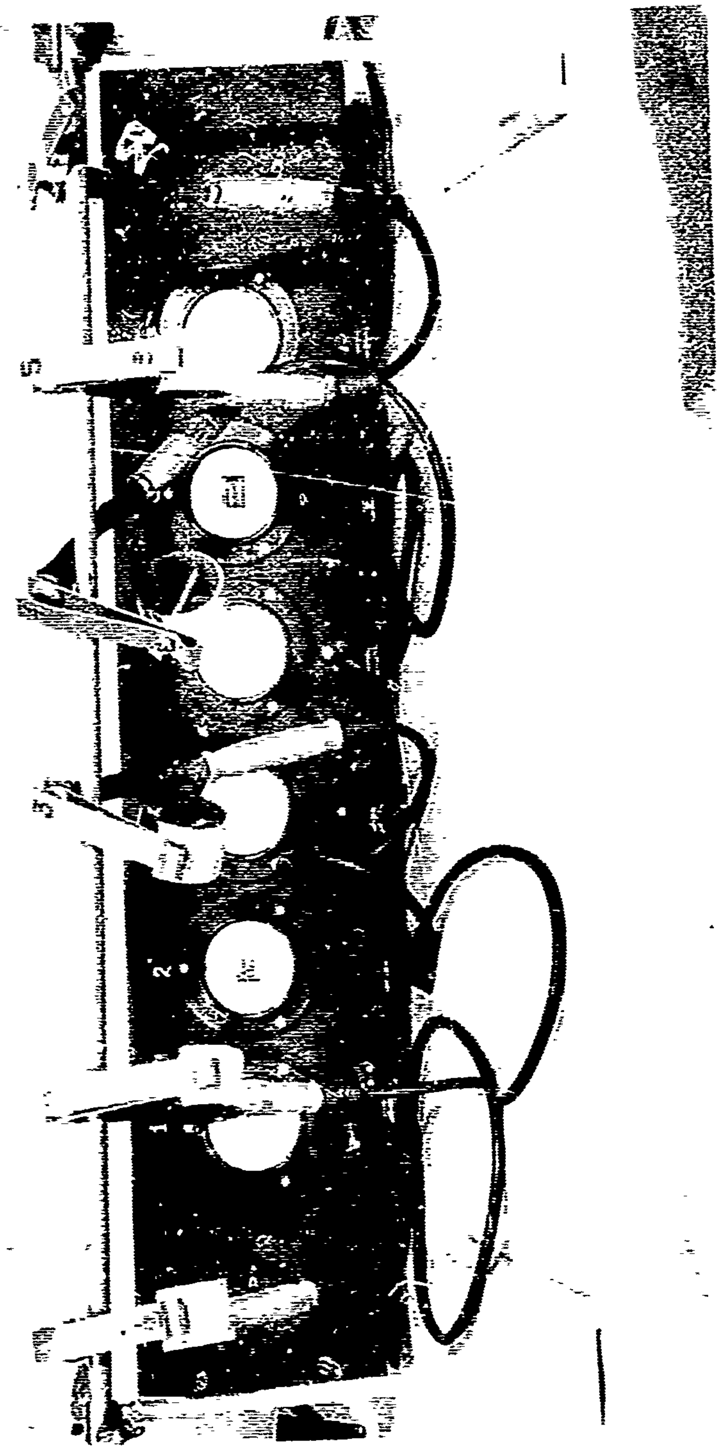
TEST RESULTS

Stripper	Lawrence Tubes HP	Lawrence Tubes λ	Drilube Tubes HP	Drilube Tubes λ	Notched C-Rings Fracture Time (hrs)	Drilube Bolts Fracture Time (hrs)	HEP Strips (R_c 51) Fracture Time	HEP Strips (Standard) Fracture Time
Stripper A	212	53.6	8.8(11)	40	1.8	1.3	783.9 min (13.1 hrs)	Did not fail 12000 min (200 hrs) (Only two strips failed)
	207	56.4	5.6(12)	36*	1.3	1.3		
			9.0(11)	42	11.6	1.6		
					34.4	2.4		
					1.9			
Stripper B	4.2	101.2	3.0(8)	long	14.5	> 7 < 100	2128.5 min (35.5 hrs)	Not tested
	6.8	104.8	8.0(6)	long	3.4	> 7 < 100		
					8.6	> 7 < 100		
					4.9	> 7 < 100		
					34.9	> 32 < 72		
					16.2	> 32 < 72		
					3.0			
					200			
Stripper C	0.23	168	**	long*	500	500	5000 min (83.3 hrs)	Not tested
	0.28	188	**	long*	500	500		
					500	500		
					500	500		

* Paint undercut

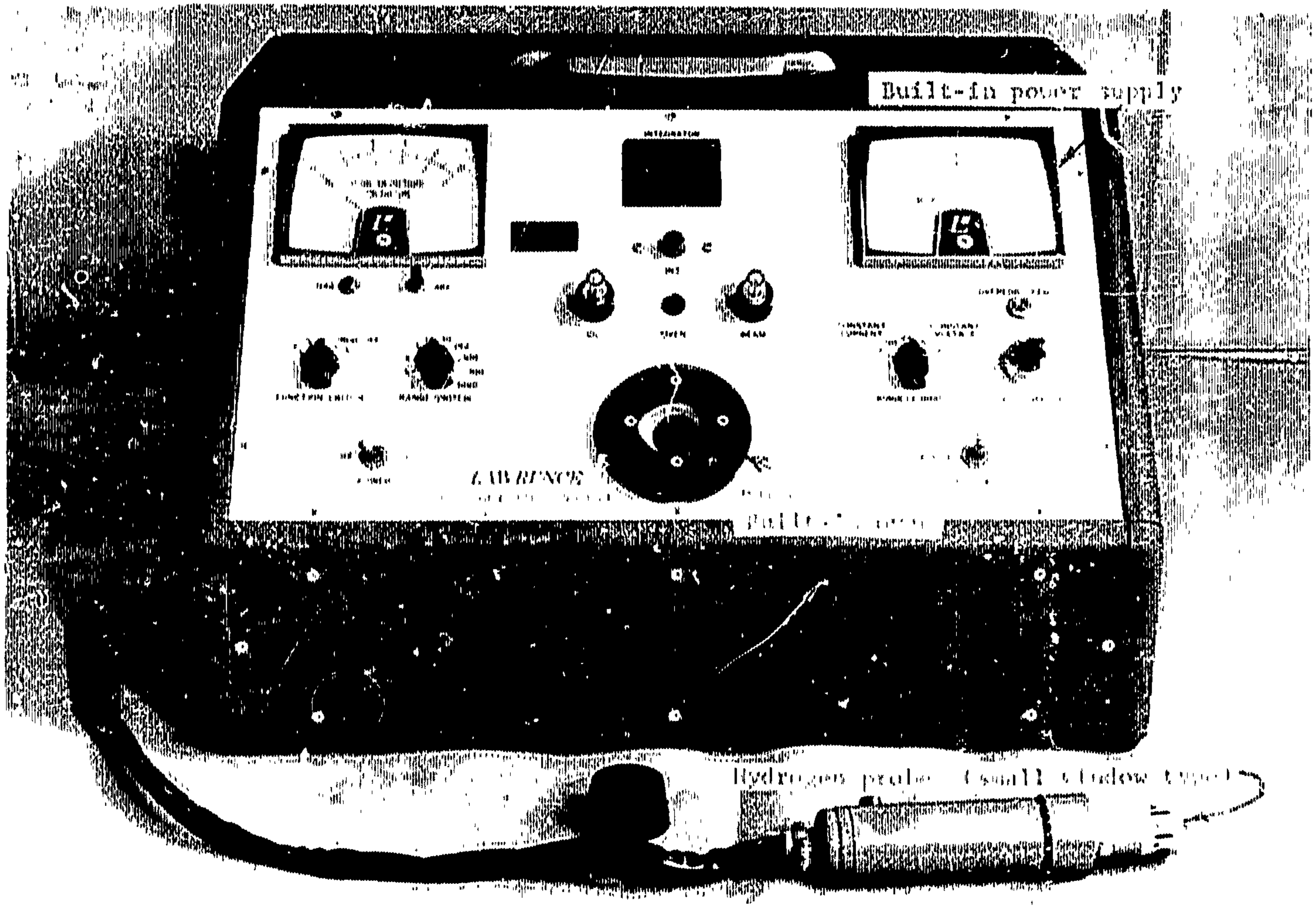
** Not measureable - very low

TABLE I



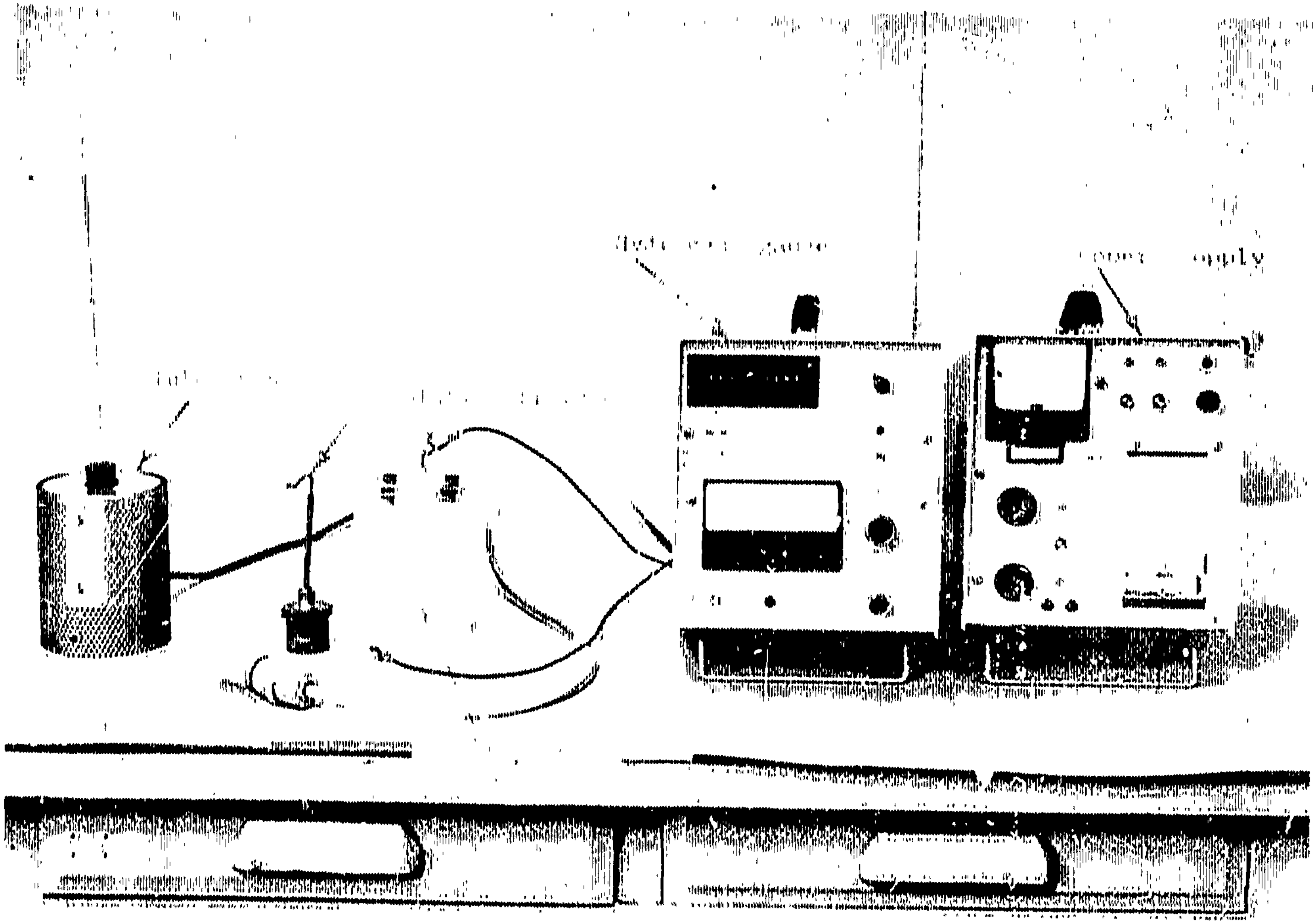
Notched C-Rings with Timers

Figure 1

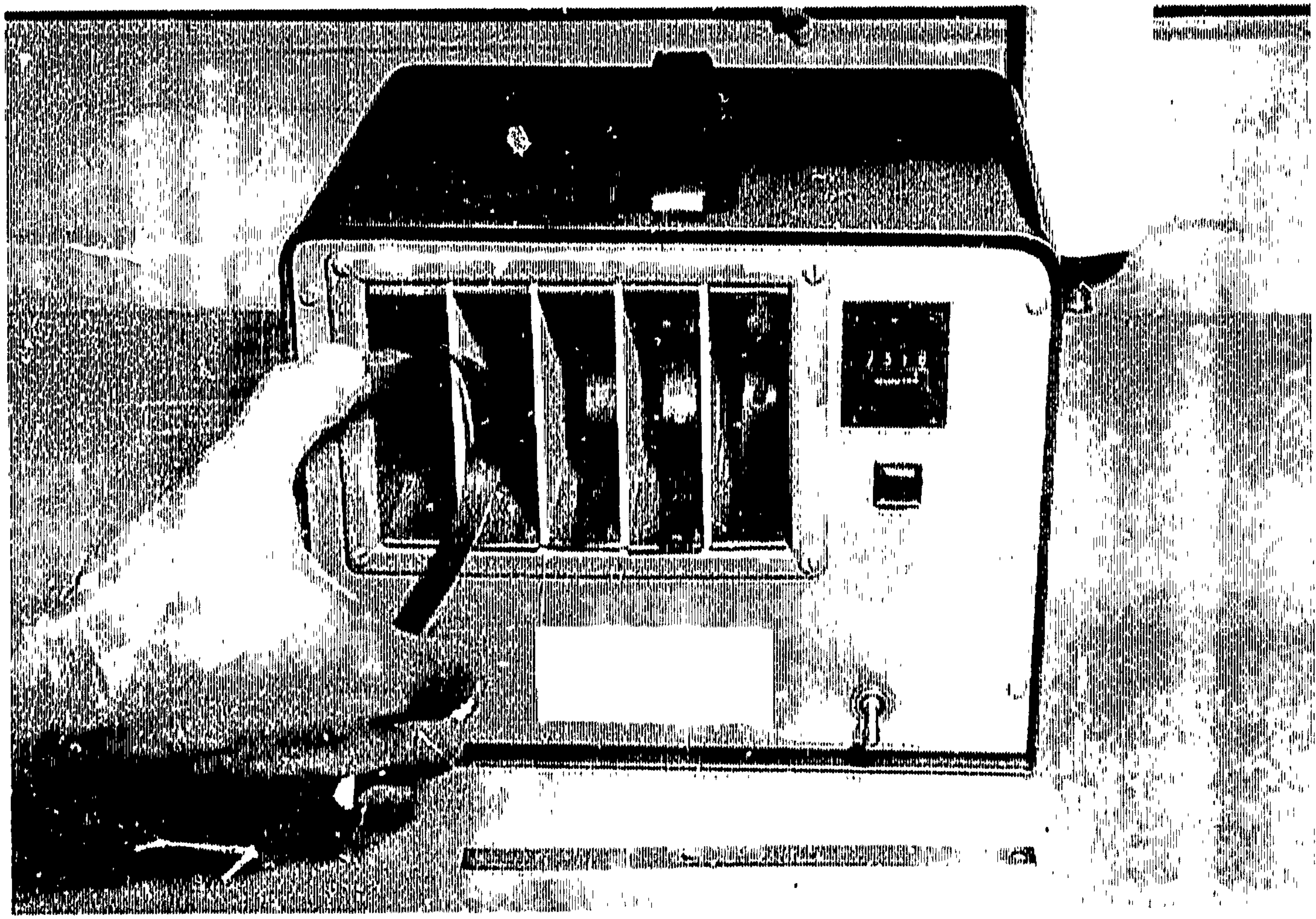


The Lawrence Hydrogen Detection Gauge

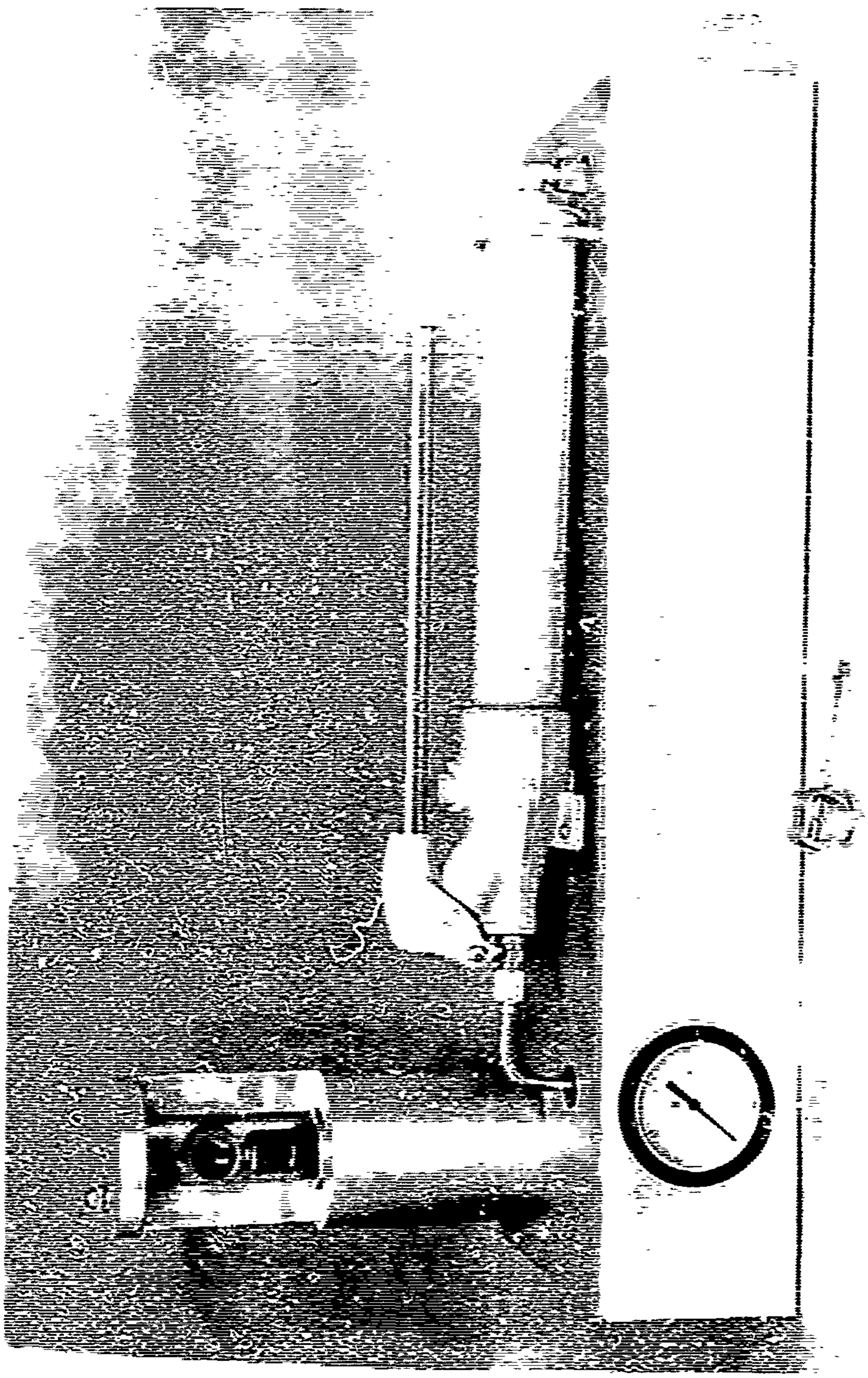
Figure 2



The Drilube Hydrogen Evaluation Gauge



The [illegible] Tester



Portable Notched Bolt Loading Device

Figure 5